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MICROSCOPIC CHARACTERIZATION OF OIL SANDS PROCESSING EMULSIONS

R.J. Mikula, V.A. Munoz, W.W. Lam

Division Report CRL 89-26(TR)
March 1989

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MICROSCOPIC CHARACTERIZATION OF OIL SANDS PROCESSING EMULSIONS

By

Randy J. Mikula*, Vicente A. Munoz** and Wendy W. Lam***

ABSTRACT

Microscopic characterization of oil sands emulsions can be important in the prediction of processing characteristics and process yields in the extraction of oil from oil sands. The size distribution of the emulsion can determine how efficiently the oil can be separated from the water and by what means: mechanically or chemically. In addition, it is possible to characterize the nature of the dispersed phase by observing fluorescence behaviour under an optical microscope or via x-ray analysis with a scanning electron microscope. In certain cases it is also possible to characterize the interface between the dispersed and the continuous phases. This paper presents results from our laboratory using microscopic techniques and illustrates their utility, not only for determining the morphology of these economically important emulsions, but also for characterizing the composition of the interface itself.

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CARACTÉRISATION PAR MICROSCOPIE DES ÉMULSIONS DE SABLES BITUMINEUX

par

Randy J. Mikula*, Vincente A. Munoz** et Wendy W. Lam***

RÉSUMÉ

La caractérisation par microscopie des émulsions de sables bitumineux peut être un facteur important lorsqu'il s'agit de prévoir les caractéristiques de traitement et le rendement des procédés utilisés pour extraire les hydrocarbures contenus dans les sables bitumineux. L'efficacité avec laquelle le pétrole peut être séparé de l'eau et la méthode utilisée pour y arriver, c'est-à-dire par voie mécanique ou chimique, peuvent dépendre de la distribution des tailles des gouttelettes dans l'émulsion. De plus, on peut caractériser la nature de la phase dispersée en observant la fluorescence par microscopie optique ou en procédant à l'analyse aux rayons X par microscopie électronique à balayage. Dans certains cas, il est possible de caractériser l'interface entre les phases dispersée et continue. Dans cette communication, on présente les résultats obtenus par analyse microscopique dans notre laboratoire, et on montre que ces techniques peuvent servir non seulement à déterminer la morphologie de ces émulsions importantes du point de vue économique, mais également à caractériser la composition de l'interface.

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INTRODUCTION

Both oil-in-water (o/w) and water-in-oil (w/o) emulsions are produced during the extraction of oil from oil sands. Prediction and prevention of loss of bitumen to the tailings stream and carry-over of water with the bitumen product is important in determining extraction process yields. Characterization of these process emulsions is crucial in understanding the fundamental aspects of their formation, breaking, and therefore their impact on extraction efficiency. The processing and upgrading of the bitumen product depends upon how efficiently the oil can be separated from the water and mineral matter and by what means: chemical, using demulsifiers; or mechanical, using centrifuges; or some combination of both. The size distribution of the process emulsions is important in the choice of separation method. For example, under industrial processing conditions, mechanical separation is often not possible for a dispersed phase size of less than about 1 μm. This limit is determined by the density difference between the dispersed and continuous phases and the residence time in the separation device. In the case of chemical separation using demulsifiers, the composition of the emulsion interface is important. A recent review on the characterization of oil-water interfaces containing finely divided solids is given by Menon and Wasan (1988).

A number of particle characterization methods such as light scattering and electrical resistance measurements have been applied to dispersions and emulsions (Groves, 1984). Other techniques include: Fourier transform infrared (FTIR), nuclear magnetic resonance (NMR), and gas chromatography/mass spectrometry (GC/MS), for the characterization of organic composition; and x-ray diffraction (XRD) and inductively coupled plasma (ICP), for the characterization of mineral and chemical composition. Electron microscopy has been used extensively for the characterization of pharmaceutical and biological emulsion systems (Donaldson et al., 1986; Rowe and McMahon, 1987), and the technique has also been applied by Zajic et al., (1981) to probe the microstructure of Athabasca oil sands. These authors used transmission electron microscopy to study replicas of freeze-fracture oil sand samples.

Recently, cryogenic scanning electron microscopy of frozen hydrated samples has been utilized, in conjunction with x-ray microanalysis,

as an analytical tool in the chemical characterization of oil sands process emulsions (Mikula, 1987&1988). In addition, the effects of oil sands minerals on emulsion stability and on extraction processes have been studied using microscopy (Mikula et al., 1988). Cryogenic preparation and the direct observation of frozen hydrated samples not only probes the morphology but also provides compositional information about the samples. The x-ray information which is available with observation of frozen hydrated samples is absent in the study of replicas.

Presently in CANMET's Fuel Processing Laboratory, both the optical and the scanning electron microscopes are interfaced to an automated image analysis system. This allows for the gathering of statistically significant quantitative information from different fields of view. This paper illustrates the capabilities of microscopy in the study of o/w and w/o oil sands processing emulsions with regard to the characterization of the dispersed phase, the continuous phase, and the interface. Both the morphological and chemical characteristics which impact on emulsion properties and therefore processing characteristics are discussed.

EXPERIMENTAL

The scanning electron microscope (SEM) used is a Hitachi X-650 equipped with both energy- and wavelength-dispersive spectrometers. An EMscope 2000 cryo-system is used to prepare the frozen hydrated emulsion samples. The procedure involves freezing the sample in a liquid nitrogen slush and keeping it frozen and under vacuum in the cryo-stage. The sample is then fractured to reveal the interior of the frozen emulsion before transferring it to the cold stage in the SEM. The cold stage in the SEM is a Hexland DN302; its temperature is maintained at ~ 90 K by an Oxford ITC4 nitrogen heat exchanger and temperature-controller unit. The energy-dispersive x-ray detector used in the cryo-stage work is a 30-mm^3 Si(Li) Tracor Northern detector coupled to the SEM and to the automated image analyser. Low electron energy (15 keV) and short acquisition times are used while accumulating x-ray spectra in the spot mode, in order to minimize beam damage and local evaporation of the sample. The details of these procedures have been described elsewhere (Mikula, 1987 & 1988).

The optical microscopy was carried out using a Carl Zeiss research microscope. The incident light system (reflected light) is equipped with two light sources: a white light source consisting of a 12 V, 100 W halogen lamp with an average brightness of 1,750 cd/cm^2; and a blue-violet light source consisting of a high-pressure mercury lamp, HBO 100 W/2, with an average brightness of 170,000 cd/cm^3. The blue light reflector uses a set of filters which provides an incident light wavelength between 450 and 490 nm. The fluorescent light from the samples passes through a barrier filter alloving only wavelengths longer than 510 nm to reach the eyepieces and camera.

The effect of sodium hydroxide (NaOH) concentration on the size distribution of the middlings has been investigated. Bitumen is separated in a bench-top simulation of oil sands extraction by the Clark hot water process at 82°C and 1 to 3 ml samples of the middlings are removed for analysis. The variation of oil droplet size with NaOH concentration (0.024 to 2.4 mg/g oil sands) has been determined by analysis of the optical microscopic images.

The effect of iron minerals on oil sands processing characteristics has also been investigated by studying field samples and by preparing a model emulsion system containing 5 mg iron (III) chloride, 225 ml water, 25 mg mineral matter, 25 g toluene, and 25 mg asphaltenes. The asphaltenes were obtained from selective extraction of oil sands samples from the Athabasca oil sands. The mineral matter was obtained from the same source, with the organic components removed by low-temperature ashing. Iron (III) chloride was dissolved in the water and mixed with the low-temperature ash solids in a blender for 60 s at low speed. The toluene and asphaltenes were then mixed in a small beaker with a spatula, added to the suspension in the blender, and mixed for 5 min. at high speed. Another emulsion was prepared without iron (III) chloride added. Afterwards, a few droplets of the emulsion samples were placed in a Neubauer hemacytometer for observation.

RESULTS AND DISCUSSION

Alberta oil sands (AOS) are known to be water-wet (Camp, 1976 & 1977) and therefore the first step in extraction of oil from oil sands is via the Clark hot water process in which oil and sand are separated after heating and the addition of caustic. The oil which does not completely separate might end up in the middlings as an o/w emulsion, or in the tailings. The tailings bitumen is generally lost, but the middlings undergoes further processing, often by flotation. The efficiency of the conventional oil sands processing by the Clark hot water process then depends upon factors such as the amount of bitumen lost to the tailings and the amount of water carried with the bitumen product. These factors are dependent upon the nature of the w/o emulsion formed in the bitumen product and the nature of o/w emulsion formed in the tailings sludge. The chemical and physical properties of the middlings, or the oil which does not initially show up in either the product or the tailings, is also important in determining the ultimate processability or processing efficiency.

The additive concentration, in this case sodium hydroxide (NaOH), is found to affect the amount of oil which can initially be separated from the sand and float to the top, as well as the droplet size of the middlings oil. The variation of mean oil droplet size of the middlings bitumen with varying NaOH concentration is measured using an image analysis system. Under blue light some of the organic components in the bitumen fluoresce and are therefore resolved more readily than when observed under white light. Figures 1a to 1d are photomicrographs of oil droplets sampled from the middlings in a simulated hot water process. Figure 1a is from the control run where no NaOH was added and shows a mean droplet size of 2.2 ± 0.1 μm. When 0.024 mg NaOH/g oil sands was added, however, the mean droplet size is decreased to 1.20 ± 0.02 μm (Figure 1b). A ten-fold increase in the concentration of NaOH added (0.24 mg NaOH/g oil sands) further decreases the mean droplet size to 0.60 ± 0.01 μm (Figure 1c). This concentration of NaOH also produced the highest yield of oil recovery in the primary flotation process. However, the trend of decreasing droplet size with increasing additive concentration is reversed when 2.4 mg NaOH/g oil sands was added. In this case, the mean droplet size increases to 3.1 ± 0.2 μm (Figure 1d).

The consequences of reagent addition to the hot water process are thus two-fold: firstly, an optimum additive concentration can be reached for maximum bitumen extraction from the oil sands; in addition, the small oil droplet size in the middlings increases the efficiency of oil separation in the middlings by induced gas froth flotation.

Figure 2 shows the SEM micrograph of a laboratory o/w emulsion prepared with oil sands bitumen and a commercial emulsifier, and identifies the locations used for x-ray analysis of the dispersed phase, the continuous phase, and the interface (Mikula, 1987). It is not possible to precisely determine the interface composition because the size of the x-ray excitation volume (approximately 1 μm^3 at 15 keV) includes x-ray signal contribution to the interface from the dispersed and the continuous phases, but useful data can still be obtained from the resulting x-ray spectra, where intensity of the x-ray signals is plotted against energy from 0 to 10 keV (shown in Figure 3a). The sulphur peak in the x-ray spectrum of the dispersed phase clearly indicates the oil component, while no peaks are seen in the spectrum of the continuous phase (Figure 3b). The broad hump is due to the bremsstrahlung background radiation. The x-ray spectrum of the interface (Figure 3c) shows a significant chlorine component which is not present in either the oil or the water phase. The chlorine contribution comes from the initial preparation of the emulsion whereby the emulsifier was dissolved in dilute HCl. Thus, the chlorine peak serves as a tracer for the distribution of the emulsifier. This technology can be used to verify the presence of clay and other mineral components at emulsion interfaces and thereby determine the importance of these components in emulsion stabilization.

The ability to characterize mineral components in these emulsion systems leads to consideration of the impact of oil sands geology and resultant mineral composition. Understanding the relationship between the mineralogy of the oil sands feed and its processability will help develop or improve methods to predict and therefore to reduce losses in process efficiency. The proportion of fine clays in the oil sands feed has been shown to be important in determining processing behaviour (Sanford, 1983; Gelot et al., 1984; Takamura and Wallace, 1986). These fine clays interact strongly with organic species and are often associated with chemisorbed organic matter on the mineral surface (Strausz, 1984; Cyr and Strausz, 1984). These chemisorbed organic species on sand and mineral components can

stabilize w/o emulsions formed in the bitumen product and help provide the bridge which leads to carry-over of water and mineral matter with the bitumen product, and loss of bitumen to the tailings. The SE micrograph of a bitumen froth from a bench-top simulation of the Clark hot water extraction process (Figure 4) shows that the clays are associated with the dispersed water phase. These clays, associated with mineral and chemisorbed organic components, can play an important role in determining the processing behaviour of an oil sands feed. The ease with which these clay-mineral and water dispersions are separated from the oil phase is a function of their size distribution, the smaller entities being much more difficult to separate.

Clays also play a role in the loss of bitumen to the tailings. In addition to losses caused by chemical interactions, these clays often form large flocs, physically trapping the bitumen and causing significant losses to the tailings. This is illustrated in the SE micrograph (Figure 5a) of a tailings sample where the bitumen is seen to be surrounded and trapped in a large clay floc. The bitumen and clay distribution is apparent from compositional analysis provided by SEM x-ray dot mapping. As shown in Figure 5b, the aluminum and silicon x-ray dot map identifies kaolinite clays in the bright regions (the potassium dot map showed no x-ray signal over the same field of view, indicating that the clays are not illites). The bitumen is indicated by the dark region, where neither aluminum, silicon, nor potassium has been detected. In this field sample, the yield of bitumen could be increased by diluting the system and increasing agitation, thereby freeing the physically trapped bitumen.

Iron minerals are thought to play a significant role in oil sands processing (Mikula et al., 1988). For example, iron hydroxides (e.g. goethite, FeO(OH)) are known for their colloidal characteristics and gel-like properties, and they, along with the carbonates, are thought to provide a bridge for interactions between certain organic components in the bitumen and the mineral/water phases (Yong and Sethi, 1978; Kotlyar et al., 1984; Schramm et al., 1985).

In oil sands processing, a final bitumen product with an average water content of about 4% indicates a "good" processing oil sand, as compared with a "poor" processing oil sand where the product might have a water content of about 8%. X-ray microanalysis of the solids in these

bitumen products showed some interesting correlations with the processing characteristics. The back scattered electron (BSE) detector of the SEM gives an image in which the brightness is proportional to the average atomic number (Z). Figures 6a and 6b are SE micrographs of solids from "good" and "poor" processing oil sands, respectively, taken with the BSE detector. The difference between the two oil sands is obvious: the "poor" processing oil sand (Figure 6b) is generally brighter and has a higher proportion of bright components which indicates a greater proportion of higher atomic number constituents; namely iron sulphides (Fe_xS_y), ilmenite ($FeTiO_3$), siderite ($FeCO_3$), etc. than the "good" processing oil sands (Figure 6a). The darker regions are silica (SiO_2) and clays.

The x-ray spectra of "poor" and "good" processing oil sands (Figures 7a & 7b) show clearly that the former has higher iron, calcium, and titanium contents than the latter. The iron minerals which are in the bitumen product in both "poor" and "good" oil sands appear to occur mainly as hydroxides and carbonates. The pyrites are probably rejected early in the processing. In fact, preliminary studies have shown that the proportion of pyrite in the bitumen product has little effect on processability compared to other iron compounds.

The effect of iron on oil sands processing was further investigated using model emulsions as well as samples from pilot and full scale oil sands extraction. Oil-in-water emulsion samples were prepared as model systems from toluene, water, asphaltenes, and mineral matter, with and without iron (III) chloride added. The emulsions were observed under the optical microscope immediately after preparation. The white light photomicrographs of the o/w emulsions with and without added iron (III) (Figures 8a & 8b) show a similar size distribution and dispersion of the oil phase.

Under high-intensity blue light, the oil phase fluoresces and the emulsion containing iron (III) is quite stable (Figure 8c), even after 5 min. This is in contrast to the case of the emulsion without added iron (III) (Figure 8d), where agglomeration of the dispersed oil is occurring after only 1 min. The stabilization of the iron-containing emulsion in this idealized case could also be due to other factors such as the chemistry of the water phase and the organic components. The investigation of these

preliminary correlations between iron content, mineral composition, and process efficiency in pilot samples and full-scale plant samples is continuing. Further development of the link between the automated image analysis system and the compositional information available from the SEM should allow quantification of iron hydroxide and iron carbonate concentrations. Since it is probably the amorphous components which are the most influential in determining emulsion stability, the information obtained from the microscopic-image analysis system represents a significant advantage over that obtained *via* x-ray diffraction.

CONCLUSIONS

The oil sands geology and resultant mineralogy of oil sands feed are important factors in determining process yields and efficiency. Significant differences in processing characteristics appear to be due to the presence of iron compounds in the oil sands. Optical and scanning electron microscopic studies have shown that iron-containing minerals have a stabilizing influence on some of the emulsions formed during bitumen extraction and contribute to the bridging which leads to carry-over of water with the bitumen product and loss of bitumen to the tailings. However, concomitant influences such as chemistry of the water phase and amount of clay fines are also important as they may operate synergistically during processing. The aim of these preliminary studies is to acquire a better fundamental understanding of emulsion properties. Microscopy offers the potential to probe important morphological properties as well as chemical composition of both the organic (*via* optical fluorescence) and mineral (*via* x-ray fluorescence) phases of these economically important emulsion systems.

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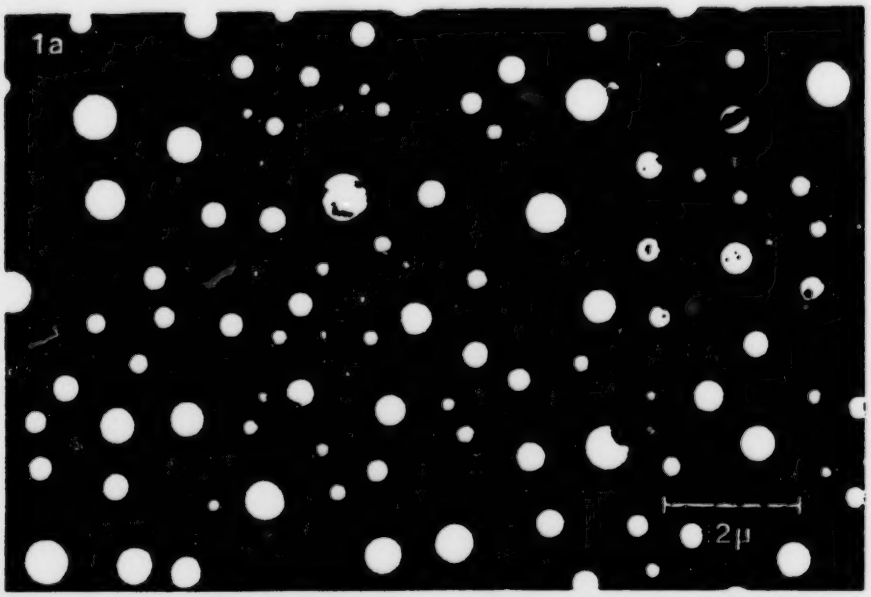
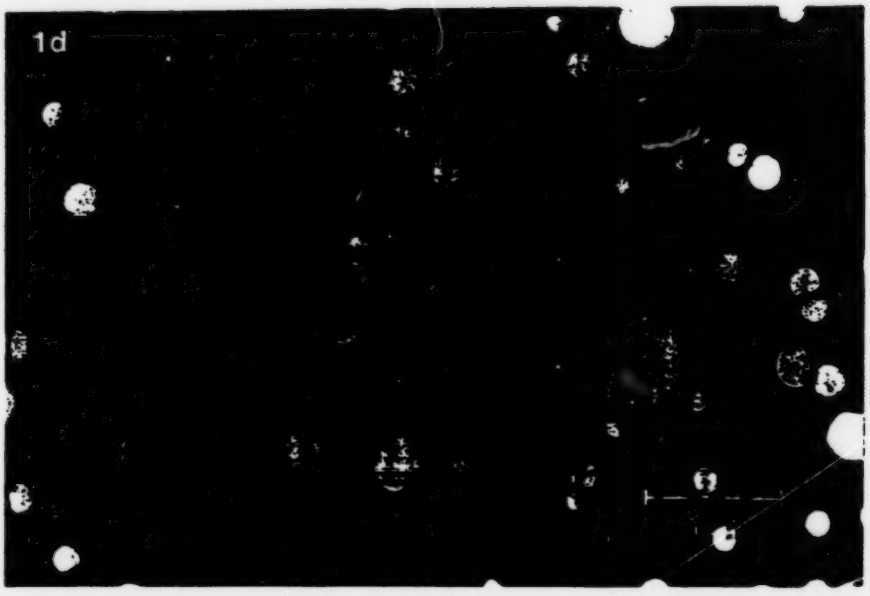


FIGS. 1a & 1b: Optical photomicrographs of oil droplets sampled from the middlings in a simulated hot water process. 1a. no NaOH added; mean droplet size = 2.2 ± 0.1 μm. 1b. 0.024 mg NaOH/g oil sands added; mean droplet size = 1.20 ± 0.02 μm.



FIGS. 1c & 1d: Optical photomicrographs of oil droplets sampled from the middlings in a simulated hot water process. 1c. 0.24 mg NaOH/g oil sands added; mean droplet size = 0.60 ± 0.01 μm. 1d. 2.4 mg NaOH/g oil sands added; mean droplet size = 3.1 ± 0.2 μm.

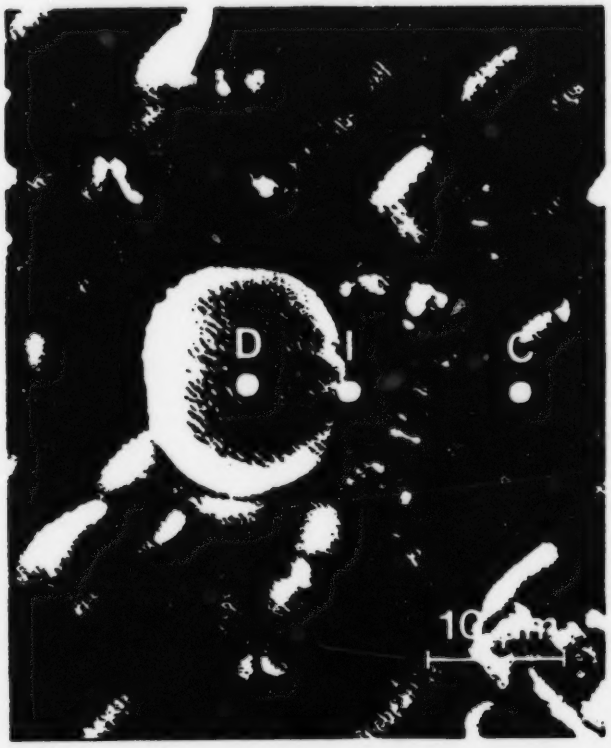


FIG. 2: SE micrograph of a laboratory o/v emulsion showing the dispersed phase (D), the continuous phase (C), and the interface (I) (adapted from Mikula, 1988).

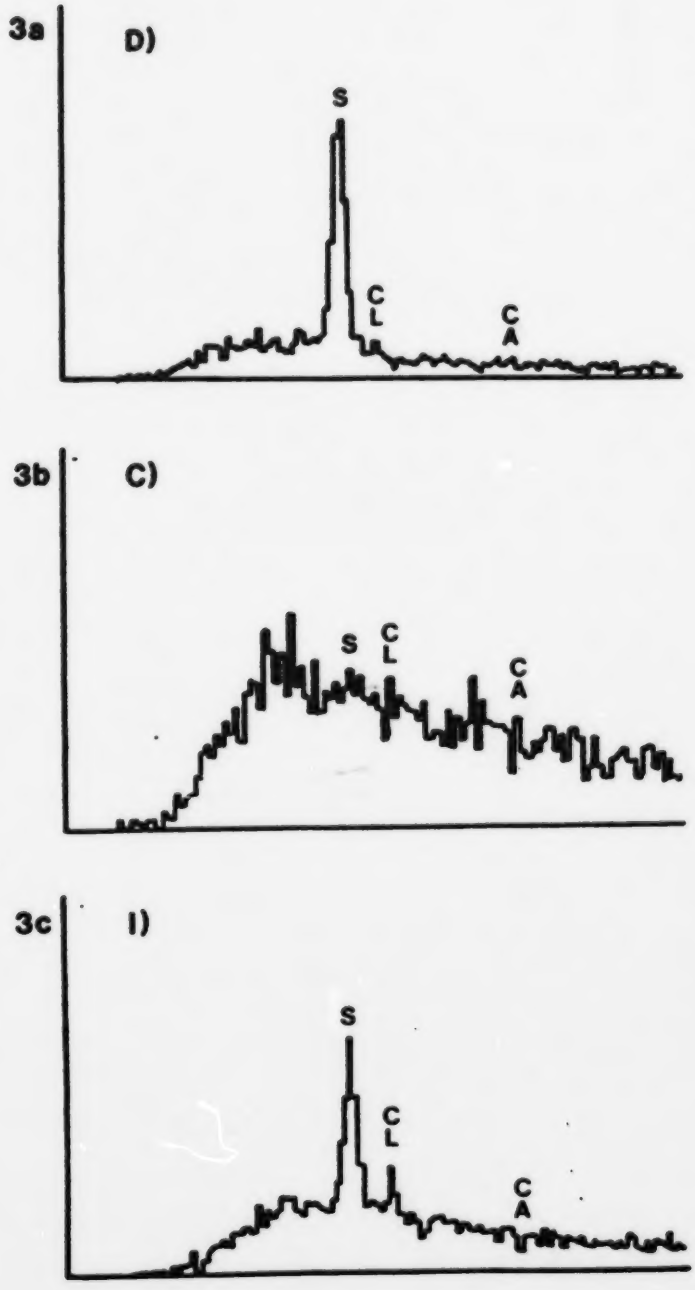


FIG. 3: X-ray spectra of an o/w emulsion (Fig. 2) corresponding to the dispersed phase D (3a), the continuous phase C (3b), and the interface I (3c).

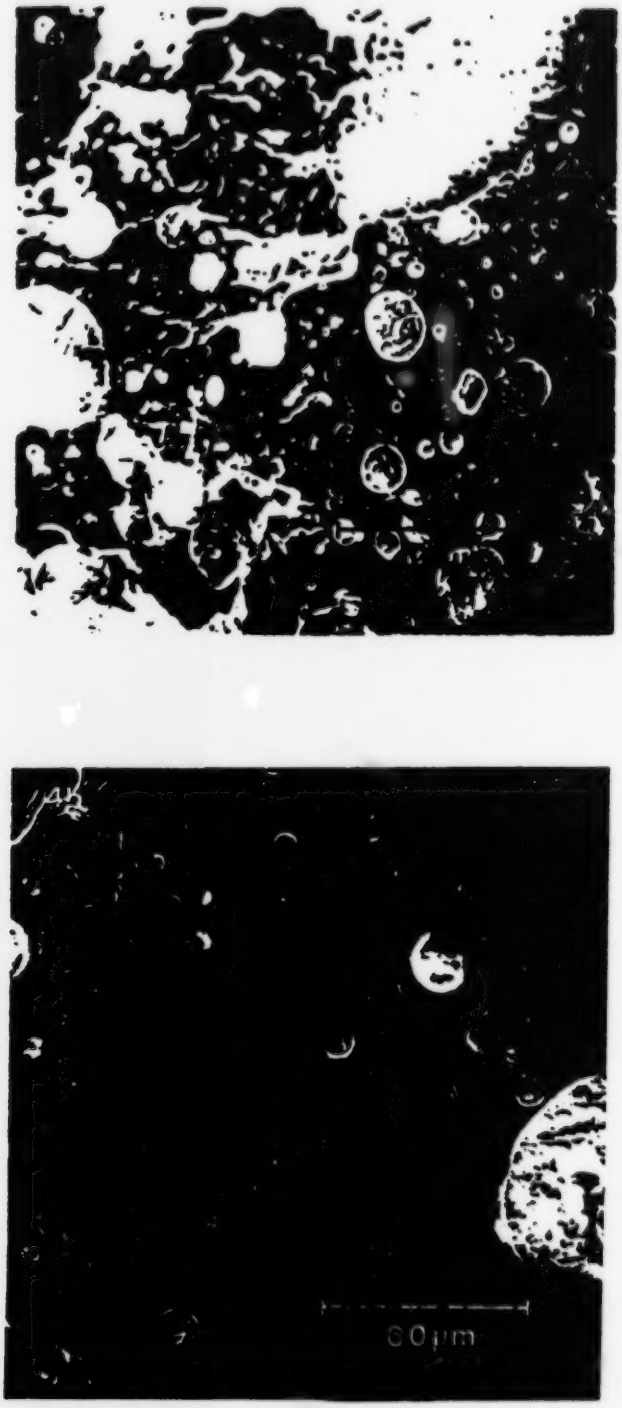
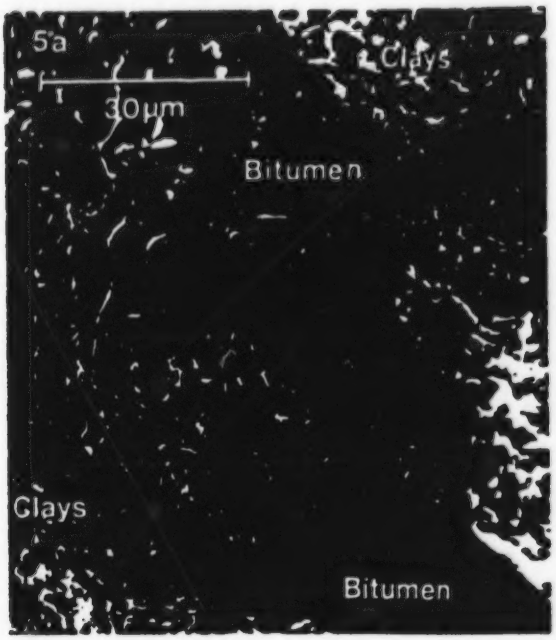


FIG. 4: SE micrograph of the bitumen froth from a bench-top simulation of the hot water extraction process shoving dispersed clays and water.



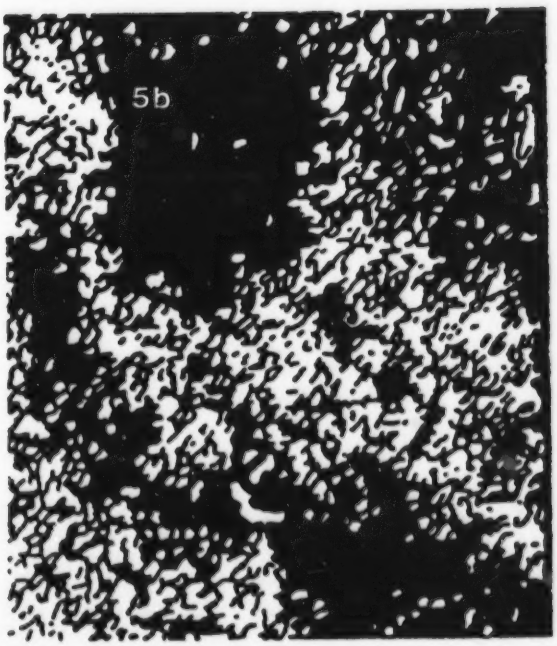
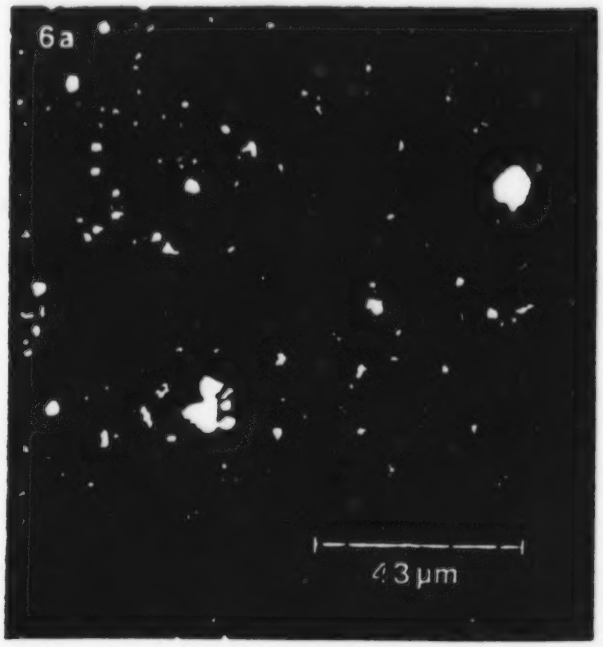


FIG. 5: SE micrograph of a tailings sample showing the bitumen being surrounded and trapped in a large fine clay floc (5a) and the accompanying x-ray dot map showing combined Al + Si intensity (5b).



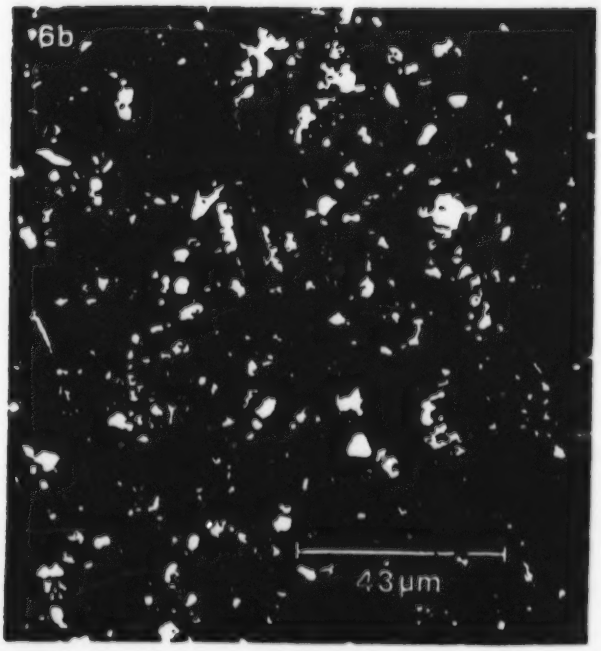


FIG. 6: SE micrographs of solids from "good" (6a) and "poor" (6b) processing oil sands taken with the backscattered electron detector.

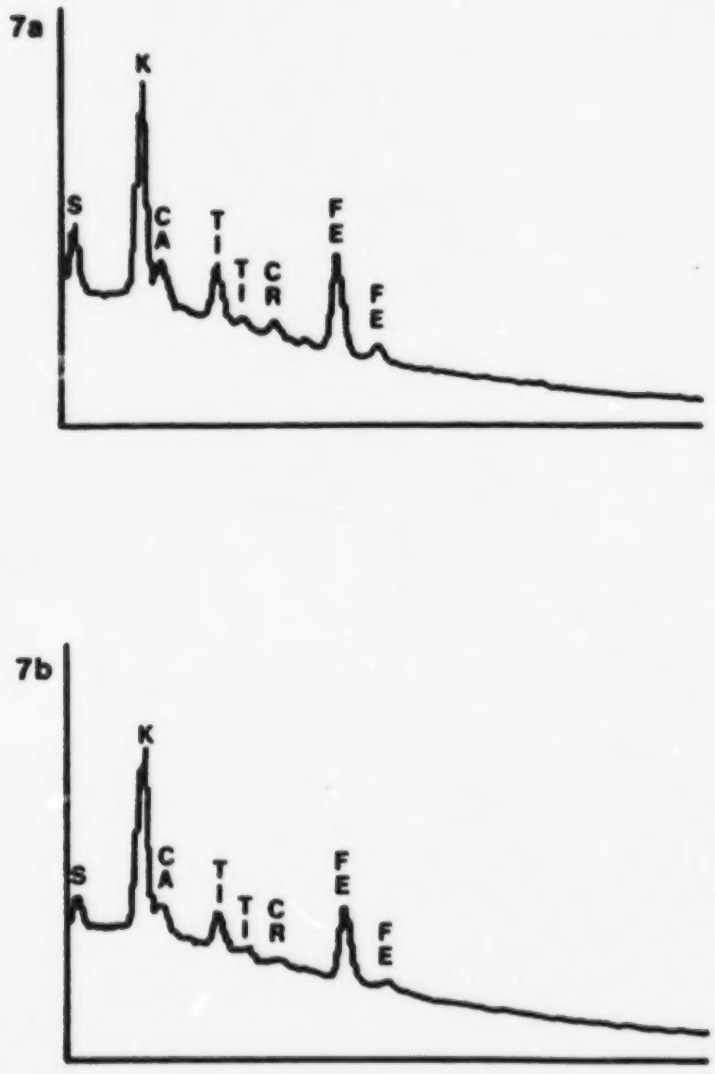
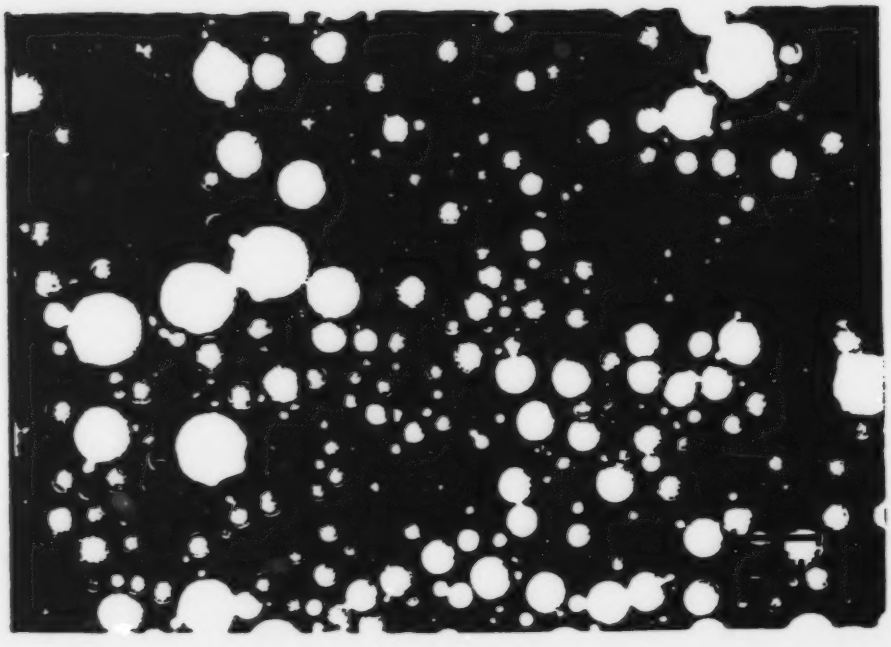
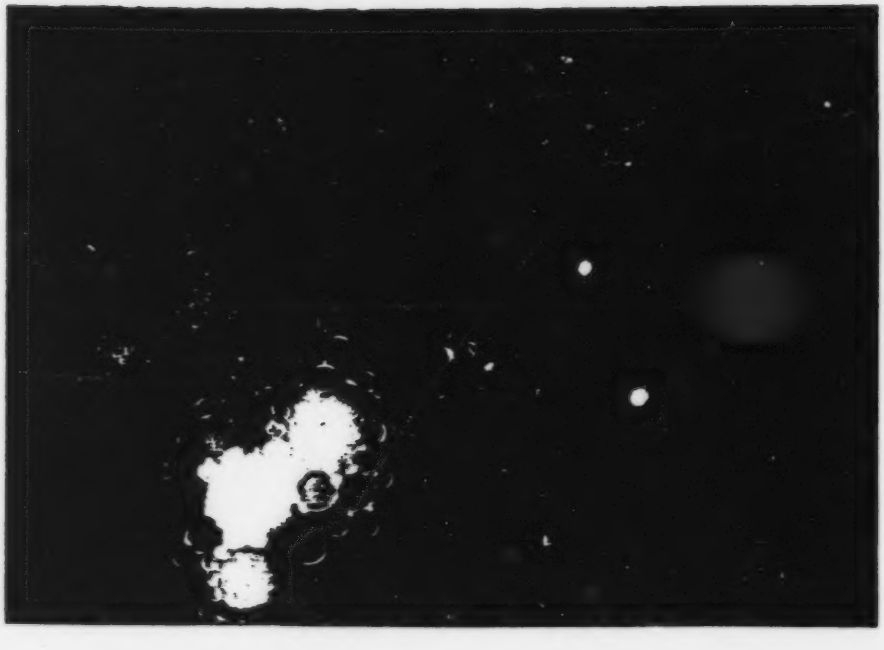


FIG. 7: X-ray spectra of toluene-extracted solids from "poor" (7a) and "good" (7b) processing oil sands.

FIGS. 8a & 8b: White light photomicrographs of a model o/w emulsion with (8a) and without (8b) added iron (III) chloride, taken immediately after preparation.





FIGS. 8c & 8d: Blue-violet light photomicrographs of a model o/v emulsion shoving that the emulsion with iron (III) chloride (8c) is stable while the one without the iron (8d) begins to coalesce.